

NASA TECHNICAL NOTE

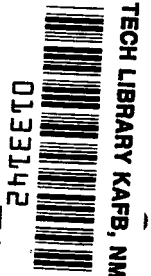


NASA TN D-6285

C.1

NASA TN D-6285

**LOAN COPY: RETI
AFWL (DOG
KIRTLAND AFB**



**EFFECT OF GRAIN SIZE ON CREEP
PROPERTIES OF A TUNGSTEN -
25-ATOMIC-PERCENT-RHENIUM -
30-ATOMIC-PERCENT-MOLYBDENUM ALLOY
FROM 1800° TO 4000° F (982° TO 2204° C)**

by Robert J. Buzzard
Lewis Research Center
Cleveland, Ohio 44135





0133142

1. Report No. NASA TN D-6285	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle EFFECT OF GRAIN SIZE ON CREEP PROPERTIES OF A TUNGSTEN - 25-ATOMIC-PERCENT-RHENIUM - 30-ATOMIC-PERCENT-MOLYBDENUM ALLOY FROM 1800° TO 4000° F (982° TO 2204° C)		5. Report Date April 1971	
		6. Performing Organization Code	
7. Author(s) Robert J. Buzzard		8. Performing Organization Report No. E-5792	
		10. Work Unit No. 120-27	
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135		11. Contract or Grant No.	
		13. Type of Report and Period Covered Technical Note	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D. C. 20546		14. Sponsoring Agency Code	
15. Supplementary Notes			
16. Abstract The stress to achieve 1 percent total creep strain at temperatures of 1800° F (982° C) to 4000° F (2204° C) was determined for powder metallurgy W-25 at. % Re-30 at. % Mo sheet. Grain sizes of 0.0014 and 0.0025 cm and a duplex structure of 0.0028 to 0.063 cm, resulted from 2-hr anneals at 2900° F (1593° C), 3600° F (1982° C), and 4000° F (2204° C), respectively. For each annealing treatment, room temperature bend ductility also was determined. The 0.0025-cm grain size material offered the best creep resistance of the three at low temperatures (below 2400° F (1316° C)) and high stresses (4 ksi (27.6 MN/m ²) and above) and was ductile at room temperature.			
17. Key Words (Suggested by Author(s)) Tungsten-molybdenum-rhenium alloy Creep properties Grain size effects on tungsten alloy Ternary alloy Duplex structure		18. Distribution Statement Unclassified - unlimited	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 27	22. Price* \$3.00

EFFECT OF GRAIN SIZE ON CREEP PROPERTIES OF A TUNGSTEN - 25-ATOMIC-
PERCENT-RHENIUM - 30-ATOMIC-PERCENT-MOLYBDENUM ALLOY

FROM 1800° TO 4000° F (982° TO 2204° C)

by Robert J. Buzzard

Lewis Research Center

SUMMARY

The stress to achieve 1 percent creep strain at temperatures of 1800° F (982° C) and 4000° F (2204° C) was determined for powder metallurgy tungsten - 25-atomic-percent rhenium - 30-atomic-percent molybdenum sheet. Test data were obtained up to 3000 hours, and isothermal plots of the data were made. Three grain sizes (0.0014 cm, 0.0025 cm, and a mixed-grain-size duplex structure of 0.0028 to 0.063 cm) were investigated. These grain sizes resulted from 2-hour anneals at 2900° F (1593° C), 3600° F (1982° C), and 4000° F (2204° C), respectively. The 0.0014- and 0.0025-centimeter grain sizes were ductile when bent at room temperature, and the 0.0014-centimeter material remained ductile at room temperature even after creep testing for a total time of 5000 hours at about 1800° F (982° C). The 0.0025-centimeter-grain-size material had the best creep resistance of the three at temperatures below 2400° F (1316° C) and stresses of 4 ksi (27.6 MN/m²) and above. The large-grained, duplex-structured material had the best creep resistance at temperatures of 2400° F (1316° C) to 4000° F (2204° C) and stresses below 4 ksi (27.6 MN/m²), but this material was brittle at room temperature.

INTRODUCTION

The development of powerful, compact nuclear reactors for space-power applications such as that described in reference 1, will require the use of materials that can withstand high temperatures and long operating times. For example, alloys that are to be used as the fuel element cladding, as structural components, or as coolant piping materials in the reactor would be expected to operate in the temperature range of about

1800⁰ F (982⁰ C) to 2400⁰ F (1316⁰ C) for times up to 50 000 hours. Under these conditions, design restraints could limit the maximum allowable creep deflection to 1 percent or less. Only the refractory metals and their alloys have the potential to withstand such severe operating conditions.

One of the candidate refractory alloys being considered for such usage is the ternary tungsten - 25-atomic-percent-rhenium - 30-atomic-percent-molybdenum alloy (W-25Re-30Mo). It is hoped that this alloy will have such desirable features as satisfactory ductility and fabricability, and in addition will have the mechanical and physical properties required by the service conditions. Some insight into the fabricability of the material, in terms of the effect of welding on the grain growth, ductility, and tensile properties of the alloy from 2000⁰ F (1093⁰ C) to 3000⁰ F (1649⁰ C) is given by Lessmann and Gold in reference 2. Data are included for both powder-metallurgy-produced and arc-cast material. This reference does not include creep data, however. Much of the creep data that have been reported for this alloy were obtained at temperatures of 2912⁰ F (1600⁰ C) and above, and are reported by Conway and Flagella in references 3 and 4. Included in reference 3 are creep data from six lots of powder-metallurgy-produced W-25Re-30Mo, with each lot having a different processing history before sintering. This reference also includes a study of first-stage creep effects as a function of stress at 1200⁰ C for 10, 15, and 20 ksi (69, 104, and 138 MN/m²). Creep and rupture isotherms at 1600⁰ C for times up to 1000 hours are also presented, along with electrical resistivity measurements up to 1800⁰ C from which the thermal conductivity of the alloy was calculated.

Still missing from the literature, however, is a determination of the creep properties of the material at temperatures below 2900⁰ F (1593⁰ C). Moreover, the effect of heat treatment and resulting grain size on this creep behavior and on the bend ductility should be established in order to realize the optimum usefulness of the material in terms of fabricability and inservice creep resistance. Therefore, it is the purpose of this study to generate such data. Consequently, the tests were conducted in such a manner as to yield 1 percent total creep data for grain sizes representing conditions of maximum high temperature creep resistance, maximum room temperature bend ductility (for formability and handling purposes), and a combination of these desirable qualities. Creep tests were run at temperatures ranging from 1800⁰ F (982⁰ C) to 4000⁰ F (2204⁰ C), with emphasis on the lower temperature range of 1800⁰ F (982⁰ C) to 2400⁰ F (1316⁰ C). The data were presented as isothermal plots.

EXPERIMENTAL METHODS

Material

The material used in this study was produced commercially by powder-metallurgy

techniques (described in ref. 5). The finished sheet measured 0.020 inch (0.51 mm) thick by 3.5 inches (89 mm) wide by 12 inches (305 mm) long. Chemical analyses of the material are given in table I. All the test specimens were made from one lot of material.

Machining of Test Specimens

Bend test specimens for room temperature bend tests consisted of the 0.125-inch (3.2-mm) wide by 1-inch (25.4-mm) long cutouts from the gage sections of the creep specimen blanks. The edges of these strips were ground with number 300 grinding paper to remove effects of shearing and machining. Although these specimens were smaller than those called for by MAB specifications, as described in reference 6, it was felt that they would be satisfactory to indicate whether the material would be ductile or brittle at room temperature as a result of various heat treatments and would thereby serve the purpose of providing "same-lot" data without sacrificing material that could be used for creep-test specimens.

Creep-test specimen blanks were warm sheared from the sheet material with the longitudinal axis of the blank parallel to the rolling direction of the sheet. The blanks were subsequently electrical discharge machined to their final shape. Slight irregularities in gage width were corrected by wet sanding with a high-speed drum sander, and final edge preparation of the gage section was achieved by hand sanding with number 300 grinding paper backed up with a steel block. All sanding was done wet and in the specimen's longitudinal direction, and the final dimensional variation along the width of the gage section was within ± 0.0005 inch (0.013 mm). The shape of the finished specimen is shown in figure 1. Tungsten wire gage marks were cemented to the specimens by use of a tungsten powder - glycerin slurry, which solidified during heat treatment and bonded the gage marks to the specimens.

Test Apparatus and Procedures

An example of the high-temperature creep-rupture testing furnaces used for annealing and creep testing is shown in figure 2. These furnaces permitted testing in a vacuum of 5×10^{-7} torr (7×10^{-5} N/m²) following initial outgassing of test specimen and furnace. The furnaces use split-type tungsten-mesh heating elements. The temperature was measured and controlled by means of a tungsten - 26-percent-rhenium/tungsten thermocouple in direct contact with the center of the specimen.

The annealing procedure followed in this program for both bend and creep test speci-

mens was to heat the test specimen at a rate such that a vacuum of at least 5×10^{-5} torr (7×10^{-3} N/m²) could be maintained. Creep test specimens were heat treated and tested in one operation; that is, they were held at the prescribed heat treatment temperature for 2 hours, and the temperature was subsequently dropped to the required test temperature. The furnace was stabilized at the test temperature for about 30 minutes before the application of the dead load. During this time, initial gage length measurements were taken. The load was then applied to the specimen by lowering a hydraulic jack weight support at a rate of about 0.010 inch (0.25 mm) per minute to eliminate the possibility of shock loading the specimen. Where step loading was required, the jack was raised just enough to remove the load from the specimen. The new load was added to the weight pan and applied to the specimen as just described. The step-loading procedure is described in the appendix. Lighter loads (6 ksi or 41.36 MN/m²) could be accommodated inside the furnace chamber, and heavier loads were applied externally through a 0.25-inch (6.35-mm) diameter O-ring seal. Elongation measurements were taken periodically until the termination of the test. These measurements were taken with a cathetometer by sighting on the gage marks applied to the specimen's gage section at a 1-inch (25.4-mm) interval. A measuring reproducibility of 0.0002 inch (0.005 mm), including operator error, was estimated for use of this instrument at this gage length.

The creep specimens were tested at temperatures of 1800^o to 4000^o F (982^o to 2204^o C), and at stresses of 40 to 0.5 ksi (276 to 3.5 MN/m²).

The bend test apparatus used in determining room temperature ductility as a function of annealing temperature was a punch and die fixture which loaded the test specimen in three-point loading. The punch consisted of a high-strength steel rod having a radius of four times the thickness of the test material (4t), and the specimen supports consisted of freely turning, 0.090-inch (2.3-mm) diameter, thoriated tungsten rods, which were backed with a steel race to prevent deflection of the rollers during load application. The supports were spaced 0.50 inch (12.7 mm) apart. The apparatus was mounted in a conventional hydraulically operated tensile testing machine. The load was applied at a rate of 1 inch (25.4 mm) per minute. All testing in this apparatus was done at room temperature.

RESULTS AND DISCUSSION

Effect of Annealing on Grain Size and Bend Ductility

The effects of annealing the W-25Re-30Mo sheet are shown in figure 3. The grain size for each anneal, as measured by the circle intercept method, is compiled in table II along with the results of room-temperature bend testing. Grain diameters range from a

uniform 0.0014 centimeter for the 2900° F (1593° C) anneal (fig. 3(b)) to a nonuniformly distributed mixture of large grains (up to 0.0630 cm) and clusters of smaller grains (about 0.0028 cm) for the 4000° F (2204° C) anneal (figs. 3(h) and (i)). This duplex structure was also observed at annealing temperatures as low as 3700° F (2038° C) (fig. 3(f)); however, the nonuniformity in grain size for this anneal was not as pronounced as that which resulted from higher temperature anneals. Although increasing grain size with increased annealing temperatures would be expected for many materials, the duplexing phenomenon was not at first anticipated in this study. This result, however, was found to compare favorably with the abnormal grain growth discussed in reference 2, wherein the duplex structure was observed after 1 hour at 3500° F (1927° C) and also for longer times (1000 hr) at temperatures as low as 2600° F (1427° C).

The 3700° F (2038° C) annealed material was brittle at room temperature, based on the failure of bend test specimens to sustain a 90° bend when tested in three-point loading over a 4t radius punch (table II). The 3600° F (1982° C), 2-hour anneal resulted in a fairly uniform grain size (0.0025 cm average grain diam, fig. 3(e)), and the material was ductile at room temperature. The photomicrographs of figure 3 show not only the grain size resulting from the various anneals, but also some of the twinning which resulted from bend testing the material at room temperature. Such twinning is common to tungsten alloys containing rhenium; however, a thorough understanding of the mechanisms relating the twinning phenomenon to ductility has not as yet been attained, and any attempt at theorizing is beyond the scope of the present study.

Although the 4000° F (2204° C) anneal yielded a somewhat brittle, duplex-structural material, it was nevertheless still considered for creep testing because of its reportedly good creep resistance (ref. 3). The 0.0014-centimeter grain size was chosen for creep testing because of its comparatively good ductility, which would imply good formability and good handling characteristics. It was also expected that this material would be the most likely to retain a stable structure after long times at temperatures of interest to the reactor design previously mentioned. The intermediate grain size (0.0025 cm) was investigated as a compromise between the larger and smaller grain size; that is, it was hoped that this material would be formable, would remain ductile enough to survive postoperational handling, and would offer an improved creep resistance as compared with the smaller grain-sized material.

Creep Test Results

The creep test results obtained in this program are summarized in table III, and typical creep curves are shown in figure 4. Figure 4(a) shows that a 2-hour anneal at 2900° F (1593° C) changes the shape of the creep curve considerably from that of the

wrought material when tested at 2000° F (1093° C) and 20 ksi (138 MN/m²). As a result of the anneal, the first stage of creep observed for the wrought material was virtually eliminated, and the subsequent linear creep rate was greatly reduced. Posttest metallographic analysis of the wrought specimen showed only slight recrystallization of the gage section. This recrystallization probably resulted from a combination of temperature and strain, since the unstrained grip section of the specimen, while exposed to the testing temperature, was still in the wrought condition.

The plots of figure 4(b) show the effects of the three anneals on the creep curves of the material when tested at 2400° F (1316° C) and 12 ksi (82.7 MN/m²). The intermediate temperature anneal of 3600° F (1982° C) resulted in the lowest creep rate and longest 1 percent creep life under these test conditions. The variation in creep life can be attributed not only to differences in steady-state creep rate of the material but also to the differences in the shape of the creep curves during the first few hours of testing. The material heat treated at 3600° F (1982° C) appears to have a suppressed first-stage of creep or incubation period before the onset of second-stage (linear) creep. This increased the time to 1 percent strain for this specimen by about 7 hours as compared with the life of the specimen heat treated at 2900° F (1593° C). The largest grain size (duplex structured) material, which was heat treated at 4000° F (2204° C), has the lowest creep resistance under these test conditions. However, when tested at 2400° F (1316° C) and 4 ksi (27.6 MN/m²) the creep resistance of this material indicated a marked improvement. These creep curves are shown in figure 4(c). The reasons for this behavior in this complex alloy are not yet fully understood, but it is evident that the larger-grained material responds differently to stress than the smaller-grained material.

The larger scale plots of figure 5 represent the overall shape of the creep curves for the small-grained material. It can be seen that the material is quite ductile at rupture under the test conditions shown and that for this small grain size the 1-percent creep life goal used in reactor design considerations represents only a small part of the total creep curve for the material.

Posttest chemical analyses of the creep test specimens (table I) showed that essentially no interstitial element contamination resulted from the creep-test environment.

Creep Life Isotherms

The isotherms of figures 6(a) to (d) show the time to reach 1-percent total creep strain as a function of applied stress at various test temperatures. The data include strain on loading as well as on any first-stage creep effects. The isotherms are shown as lines drawn through the raw data points. The data points shown as open symbols represent tests which were not run to 1-percent total strain because of the time factor

involved; rather, the linear creep rate was established and the time to reach 1 percent was extrapolated by extension of this linear portion of the creep curve. This procedure was thought to be a reasonable approach to the problem of obtaining long-time data points for this study, since the creep curves remain linear, or as nearly linear as is possible to detect by existing testing techniques, through creep strains of several percent as was shown in figure 5.

Figure 6(a) shows that the slope of the isotherms for the small grained material increases as the test temperature increases. The 2400° F (1316° C) test data for both the small and the intermediate-grain-size material (figs. 6(a) and (b)) show also that the individual isotherms curve downward at the lower stresses and longer test times. The test conditions at which such curvature would occur for the 1800° F (982° C) isotherm are not known. However, the longest test of this study was used to obtain the 1800° F (982° C) isotherm (for the small-grain-size material), and the error involved in projecting this isotherm to 50 000 hours would be expected to be less than that for the higher temperature data where such curvature was observed after relatively shorter term tests. The plots of the 2400° F (1316° C) and 2500° F (1371° C) data (fig. 6(c)) indicate that the isotherms for the large-grained, duplex-structured material apparently maintain their linearity for a longer time than do the isotherms for the smaller-grained materials of figures 6(a) and (b). Also, the 4000° F (2204° C) isotherm in figure 6(c) appears to be nearly parallel to the lower temperature plots, indicating a fairly stable stress-sensitivity of this material at the higher temperatures as compared with the other two materials (figs. 6(a) and (b)).

Also shown in figure 6(c) are data points from the study of reference 3, wherein six different processes were used to fabricate the sheet material of that study. The scatter in these data indicates the sensitivity of this material to processing history.

The 1800° F (982° C) and 2400° F (1316° C) isotherms for the various grain sizes were replotted in figure 6(d) to facilitate comparison of the 1-percent creep lives of these materials. The isotherms for a test temperature of 2400° F (1316° C) indicate that increased grain size results in better long-term creep resistance for this ternary alloy. This difference in creep life is less pronounced at test temperatures of 1800° F (982° C) for the 0.0014- and the 0.0025-centimeter grain sizes.

Posttest Examination

In addition to the metallographic examinations of material before creep testing, chemical analyses and microscopic examination of specimens were conducted after creep testing to aid in determining creep mechanisms and material structural changes, if any, as a result of creep testing. Posttest interstitial analyses are reported in table I, and

no significant contamination is indicated. Photomicrographs of test specimens representative of each grain size are shown in figure 7. In figure 7(a), the 0.0014-centimeter-grain-size specimen had been creep tested at 2200° F (1204° C) and 20 ksi (138 MN/m²) for a total creep strain of 2.3 percent. Evidences of grain boundary separation can be observed even at this low total strain. The photomicrograph in figure 7(b) shows a great amount of grain boundary separation for the same grain size material strained to 42 percent after 166 hours at 2400° F (1316° C) and 12 ksi (82.7 MN/m²). The creep curve for this specimen was shown in figure 5. Grain boundary separation is also evident in the 0.0025-centimeter-grain-size material tested at 2400° F (1316° C) for various stresses resulting in 5.9 percent total strain in 1500 hours (fig. 7(c)) and in the large grain size material tested at 4000° F (2204° C), 3005° F (1651° C), and 2600° F (1427° C) at various stresses for 567 hours and a total strain of about 7 percent. Based on these observations, it would appear that grain boundary sliding is the prime mode of failure of the W-25Re-30Mo material at 2200° F (1204° C) and above.

Specimens that were bend tested at room temperature after the creep testing at elevated temperatures showed evidence of twinning. In the largest-grained material, this twinning was audible during the room-temperature bend testing.

Creep testing appeared to have embrittled the large-grained material considerably, as this material deflected only slightly before snapping. One specimen of the intermediate grain size material was ductile after straining to about 1½ percent in 280 hours at 1800° F (982° C); however, another specimen broke after a bend angle of about 30° after creep testing to about 6 percent strain in 1500 hours at 2400° F (1316° C). The small-grained material (0.0014 cm average grain diam) remained ductile even after creep testing for very long times. In fact, a 0.0014-centimeter-grain-size specimen was used to conduct the longest test in this program. It was run for 5325 hours: at first at 1925° F (1052° C) for 533 hours and then at 1800° F (982° C) for the remainder of the test time. Total strain on the specimen was 1.92 percent. The specimen was cut in half for posttest chemical analysis, metallographic examination, and bend testing. When bent over a 4t radius at room temperature to the limit of the bend test equipment, this specimen exhibited a 125° bend angle (after springback) with no sign of failure. The bent segment of the specimen is shown in figure 8(a). Figure 8(b) shows the grain structure of the test section; there is a small amount of noticeable grain boundary separation as a result of test conditions, again indicating grain boundary sliding. Also, the grain size appears to have remained stable during the test.

Observations of the microstructures for the various grain sizes and testing conditions were made using the electron microscope, but no unusual features (such as sub-grain formation) were observed even in the large grained material at magnifications up to ×160000.

Activation Energy for Creep

The activation energy for creep for the W-25Re-30Mo alloy was obtained from a computer solution using multiple linear regression and an equation of the form

$$\dot{\epsilon} = A\sigma^n e^{-Q/RT}$$

where

$\dot{\epsilon}$ strain rate, in./in./sec

σ applied stress, psi (N/m^2)

R gas constant, $1.987 \text{ cal}/(\text{g-mole})(\text{K}) = 8.314 \text{ J}/(\text{g-mole})(\text{K})$

T temperature, K

A, n constant

Q activation energy for creep, cal/g-mole (J/g-mole)

Solution of the equation yielded values for A, n, and Q.

The activation energy value was used as a factor in relating strain rate and test temperature to facilitate the plotting of this combined variable against corresponding test stresses in the manner shown in figure 9. The slope of such a plot indicates the stress dependence of the creep rate, normalized for temperature. An initial plot of this type, using an activation energy value based on data from all the creep tests run in this program, indicated a similarity in slope for all three grain sizes at stresses above 6 ksi (41.4 MN/m^2) and below 40 ksi (276 MN/m^2). The activation energy was recalculated using only the data from this stress regime, since a similarity in the sensitivity of creep rate to applied stress for the different grain sized materials of this study implies a corresponding similarity in creep mechanism and possibly an activation energy for creep common to this range of test variables. This stress range is further defined in relation to test temperature, for the stress regime just described was also the 1800° to 2400° F (982° to 1316° C) temperature range of this study. For ease of further discussion, this stress-temperature regime will be referred to as range 2 in figure 9. In terms of the absolute melting point of this alloy, the temperature range described in range 2 is about 0.39 to 0.50 T_m , where T_m is approximately 2910° C for the W-25Re-30Mo alloy. Recalculation of the activation energy for creep, based on data from range 2, resulted in a value of 94.7 kilocalories per gram mole (396.5 kJ/g-mole). This value agrees with the value of 90 kilocalories per gram mole (376.8 kJ/g-mole) for creep in tungsten at 0.40 to 0.65 T_m reported by Robinson and Sherby in reference 7. Using this new value for Q, the stress dependence of the temperature compensated minimum creep

rate was again determined, and the least squares lines were fit through the data points of range 2 for each grain size. Least squares lines were also fit through the remaining data points for each grain size (i. e. , range 1 data) as shown in figure 9. The least squares lines for the smaller, more stable grain sized materials intersected at 8 ksi (55.1 MN/m^2), which was chosen as a more definitive dividing line between the two stress-temperature ranges. The line through the data for the duplex grained material was continuous through both ranges. Although the formulation of such a plot and its subsequent interpretation may be quite subjective, as pointed out by Gifkins in reference 8, the data of this plot nevertheless do fall into a pattern that has been observed for other metals and which may provide some insight into the creep behavior of the complicated alloy of this study. For example, the effect of grain size on the creep rate of this alloy at low stresses (range 1 of fig. 9), is that the creep rate decreases with increasing grain size, and the creep rate is nearly proportional to the stress for the 0.0014- and 0.0025-centimeter grain sizes. This could indicate diffusional creep without dislocation motion, as described by Sherby and Burke (ref. 9); however, when considering the relatively low test temperatures (near $0.5 T_m$) used in testing the smaller grain size materials in range 1 and the grain boundary separation observed during post-test metallographic analysis of this material, it is more likely that the mechanism controlling creep in range 1 of this study is grain boundary sliding. There appears to be little if any effect of grain size on creep rate in range 2, and the data approximate a three-power law stress behavior ($n = 3.68$ for the uniform grain sizes and 3.79 for the duplex-grained material). This behavior could indicate that the controlling creep mechanism in range 2 is a dislocation glide process involving the dragging of solute atoms along with dislocations, as is also discussed in reference 9. However, any attempt to define the exact mechanism controlling the creep characteristics of this alloy is well beyond the scope of the present study.

CONCLUDING REMARKS

Based on the results of this study, it would appear that, for practical applications of W-25Re-30Mo material at temperatures up to 2400° F (1316° C), an annealing treatment of 3600° F (1982° C) would provide the best creep resistance for the material while maintaining room temperature ductility (as measured by the material's ability to undergo a 90° bend over a $4t$ radius). After higher temperature anneals, the material experiences duplex structuring and becomes embrittled when bent at room temperature. For example, the very high temperature (4000° F (2204° C)), 2-hour anneal causes significant preferred grain growth resulting in a mixed grain structure consisting of 0.0028 and 0.063 centimeter grains. Although the alloy is most creep resistant in this

condition when tested at temperatures above 2400° F (1316° C) and stresses below 4 ksi (27.6 MN/m²), it is brittle before testing and it becomes even more embrittled as a result of exposure to these creep testing conditions. This behavior should be kept in mind when considering the use of this ternary alloy in the welded condition, for the weld-affected zone would be expected to have a structure akin to the one just described.

It should also be emphasized that the annealed behavior described in this study may vary with different lots of material. For example, although duplexing observed in this study appeared to begin above 3600° F (1982° C), this type of behavior was observed to occur at lower temperatures and times in the study of Lessmann and Gold (ref. 2). This suggests that variations in the material's processing history, or lot-to-lot differences in material, will affect the annealing conditions necessary to cause duplexing. Furthermore, the referenced study shows duplex structuring after very long times at low temperatures (e. g. , 1000 hr at 1427° C), indicating that duplexing is also affected by time-temperature conditions.

In view of the above mentioned factors, it would appear advisable for the user of this alloy to check the response to annealing conditions for his particular lot of material should the factors just discussed be of importance to the material's application.

Based on the results of this study, the safest annealing treatment in terms of maintaining room temperature ductility after exposure to about 2400° F (1316° C) operating temperatures would be 2 hours at 2900° F (1593° C); however, this would result in a lower creep resistance than materials subjected to higher temperature anneals.

CONCLUSIONS

The following conclusions were drawn from this investigation, based on an annealing study and on bend and creep tests run on powder-metallurgy-produced, 0.020-inch (0.51-mm) thick tungsten - 25-atomic-percent-rhenium - 30-atomic-percent-molybdenum (W-25Re-30Mo) sheet. Bend tests were run at room temperature using three-point loading over a 4t radius. Creep tests were conducted in vacuum over the temperature range of 1800° F (982° C) to 4000° F (2204° C), on material in the as-fabricated (wrought) condition and after 2-hour anneals at 2900° F (1593° C), 3600° F (1982° C), and 4000° F (2204° C) which resulted in grain size of 0.0014 centimeter, 0.0025 centimeter and a duplex structure of 0.0028 and 0.063 centimeter, respectively. Isothermal plots for 1-percent total creep strain at 1800° F (982° C) and 2400° F (1316° C) were generated in this study.

1. Annealing for 2 hours at 3600° F (1982° C) resulted in the most creep-resistant material of this study, based on intermediate temperature (2400° F (1316° C) and lower) low stress (4 ksi (27.6 MN/m²) and lower) creep tests. The material was ductile at

room temperature both before creep testing and after creep testing to low strains (about $1\frac{1}{2}$ percent); however, it was very brittle at room temperature after creep testing to about 6-percent strain at 2400° F (1316° C).

2. Annealing for 2 hours at 2900° F (1593° C) yielded the least creep-resistant material; however, this material remained ductile at room temperature (based on 4t bend tests) after creep testing to about 2 percent total strain at 1800° F (982° C) and 1925° F (1052° C) for a 5300-hour total test time.

3. Annealing for 2 hours at 3700° F (2038° C) and above resulted in duplex structuring and in room temperature embrittlement of the W-25Re-30Mo alloy, based on 4t bend test data.

4. Annealing for 2 hours at 4000° F (2204° C) yielded a highly duplex-structured material which had the best creep resistance of this study at low stresses (4 ksi (27.6 MN/m^2) and lower) and temperatures above 2400° F (1316° C).

5. Recrystallization improved the creep resistance of the material, as compared with the as-fabricated (wrought) condition.

6. The activation energy for creep was calculated to be 94.7 kilocalories per gram-mole (396.4 kJ/g-mole) for the 1800° F (982° C) to 2400° F (1316° C) temperature range.

7. Grain boundary sliding appears to be the prime mode of failure of the W-25Re-30Mo alloy at creep test temperatures of 2200° F (1204° C) and above.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 5, 1971,
120-27.

APPENDIX - STEP-LOADING OF W-25Re-30Mo CREEP SPECIMENS

The term step loading refers to the creep testing of a specimen under various stress or temperature conditions, so that several creep rate versus stress (or temperature) data points can be obtained from a single test specimen during a single furnace run, thereby conserving both test specimen material and test preparation time. The purpose of this appendix is to describe the method of step loading and data analysis as applied to the present study.

When step loading was used in this study, the secondary loading stresses and temperatures were not allowed to exceed the ranges used previously for the creep testing of specimens under single load-temperature conditions. This precaution was taken so that all testing would be performed under test conditions wherein the creep rate for each step would represent the minimum creep rate that would be expected for a virgin specimen tested under the stress-temperature conditions of that step. A value of 0.07 percent strain, representing the average extension on loading, was accounted for when calculating the 1 percent total creep life for each step. This value was based on data obtained by having run other specimens under similar test conditions. This value is shown as ϵ_0 in the schematic creep curve of figure 10. In this study, each step was run to the full 1-percent total strain where practical from a time standpoint (i. e. , <1000 hr). Where this was not practical (as represented by the $\dot{\epsilon}_3$ segment of fig. 10), time to reach the 1 percent total strain, inclusive of strain on loading, was calculated using the creep rate over 0.93 percent strain (1.00 - 0.07 percent loading strain).

Applicability of this procedure was further verified by running one of the final steps at the same test temperature and stress as that used for a single specimen and comparing step results with actual single specimen test results as shown schematically in figure 10. Consistency of results was also verified by alternating the stress for each step between large and small stresses and checking the linearity of the resulting stress against 1-percent creep life isotherm for the series of runs.

REFERENCES

1. Davison, Harry W.: Preliminary Analysis of Accidents in a Lithium-Cooled Space Nuclear Powerplant. NASA TM X-1937, 1970.
2. Lessmann, G. G.; and Gold, R. E.: The Weldability of Tungsten Base Alloys. Welding J. Res. Suppl., vol. 48, no. 12, Dec. 1969, pp. 528-S-542-S.
3. Anon.: AEC Fuels and Materials Development Program. Rep. GEMP-1012, Pt. I, General Electric Co., Mar. 31, 1969.
4. Conway, J. B.; and Flagella, P. N.: Creep-Rupture Data for the Refractory Metals to High Temperatures. Rep. GEMP-685, General Electric Co., Mar. 1969.
5. Anon.: High-Temperature Materials and Reactor Component Development Programs. Vol. 1-Materials. Rep. GEMP-334 A, General Electric Co., Feb. 25, 1965.
6. Marvel, Carl S.: Evaluation Test Methods for Refractory Metal Sheet Material. Rep. MAB-192-M, Materials Advisory Board, National Research Council, Apr. 22, 1963.
7. Robinson, S. L.; and Sherby, O. D.: Mechanical Behavior of Polycrystalline Tungsten at Elevated Temperature. ACTA Met., vol. 17, no. 2, Feb. 1969, pp. 109-125.
8. Gifkins, R. C.: Transitions in Creep Behaviour. Materials Sci., vol. 5, no. 2, Feb. 1970, pp. 156-165.
9. Sherby, Oleg D.; and Burke, Peter M.: Mechanical Behavior of Crystalline Solids at Elevated Temperature. Rep. SU-DMS-67-33, Stanford Univ., Aug. 1, 1967.

TABLE I. - CHEMICAL ANALYSES OF POWDER

METALLURGY TUNGSTEN - 25-ATOMIC-

PERCENT RHENIUM - 30-ATOMIC-

PERCENT MOLYBDENUM SHEET

Element	Initial concentration ^a		Post creep test concentration ^a wt. %
	at. %	wt. %	
Tungsten	Bal.	Bal.	-----
Rhenium	24.88, 24.89	29.56, 29.57	-----
Molybdenum	30.17, 30.64	18.30, 18.59	-----
Carbon	-----	0.0009, 0.0012	0.0005, 0.0011
Oxygen	-----	0.0017, 0.0018	0.0012, 0.0025
Nitrogen	-----	0.0006, 0.0010	-----

^aDuplicate analyses were obtained.

TABLE II. - GRAIN SIZE AND ROOM TEMPERATURE

4-T BEND ANGLE FOR TUNGSTEN - 25-ATOMIC-

PERCENT RHENIUM - 30-ATOMIC-PERCENT-

MOLYBDENUM POWDER METALLURGY

SHEET, AS FUNCTION OF

2-HOUR ANNEALS

Annealing temperature		Grain size, cm	4-t Bend angle to fracture at room temperature, deg
°F	°C		
2900	1593	0.0014	>130
3200	1760	.0014	>130
3400	1871	.0018	>130
3600	1982	.0025	>120
3700	2038	0.0028 to 0.0041	30
3800	2093	0.0028 to 0.010	42
4000	2204	0.0028 to 0.063	30

TABLE III. - CREEP DATA FOR 0.020-INCH (0.51-mm) THICK POWDER

METALLURGY TUNGSTEN - 25-ATOMIC-PERCENT - RHENIUM -

30-ATOMIC-PERCENT MOLYBDENUM SHEET

2-Hour annealing temperature		Stress		Test temperature		Time to 1 percent strain, hr	Creep rate, percent/hr
$^{\circ}\text{F}$	$^{\circ}\text{C}$	ksi	MN/m ²	$^{\circ}\text{F}$	$^{\circ}\text{C}$		
----	----	20	138	2000	1093	7.3	0.06
2900	1593	20	138	2200	1204	19.0	0.05
		20		2200	1204	13.3	.066
		20		2000	1093	217	.0045
		^a 20		1925	1050	533	.00161
		25	172	1800	982	2 830	.00035
		15	104	1800	982	^b 17 712	.000053
		12	82.7	2400	1316	11.5	.0745
		15	104	2200	1204	47	-----
		12	82.7	2200	1204	96.7	.0097
		4	27.6	2400	1316	132	.0071
				2600	1427	24.3	.038
				2500	1371	65.0	.0149
				2400	1316	133	.00712
		{ 1	6.9	2600	1427	92.5	.00917
		{ 2	13.8	2500	1371	108	.00925
		{ 2	13.8	2400	1316	442	.00213
		{ 6	41.4	2400	1316	69.7	.0135
		{ 1.2	8.3	2400	1316	750	.00124
		.5	3.4	2850	1566	51.0	.019
		.5	3.4	2750	1510	^b 100	.0098
		.5	3.4	2500	1371	^b 984	.001
3600	1982	40	276	1800	982	180	0.0029
		25	172	1800	982	^b 3 400	.0003
		12	82.7	2400	1316	18	.055
		10	69.0			26.7	.038
		4	27.6			283	.00355
		2	13.8			806	.00118
		3	20.7			^b 453	.00221
		6	41.4			109	.00912
		6	41.4	2240	1227	830	.00108
		10	69.0	2240	1227	423	.00225
		20	138	2240	1227	30.0	.033
4000	2204	4	27.6	2400	1316	^b 283	0.003
		2	13.8	2500	1371	^b 1 260	.000769
		12	82.7	2500	1371	3.5	.286
		12	82.7	2400	1316	10.0	.1
		1.2	8.3	3000	1649	116	.00862
		1.2	8.3	2650	1454	^b 5 750	.000174
		.69	4.8	4000	2204	18.6	.055
		1.2	8.3	4000	2204	1.2	.855
		1.2	8.3	3005	1651	^b 341	.00292
		1.2	8.3	2600	1427	^b 4 320	.00023

^a Step loading of one specimen is indicated by {.^b Estimated.

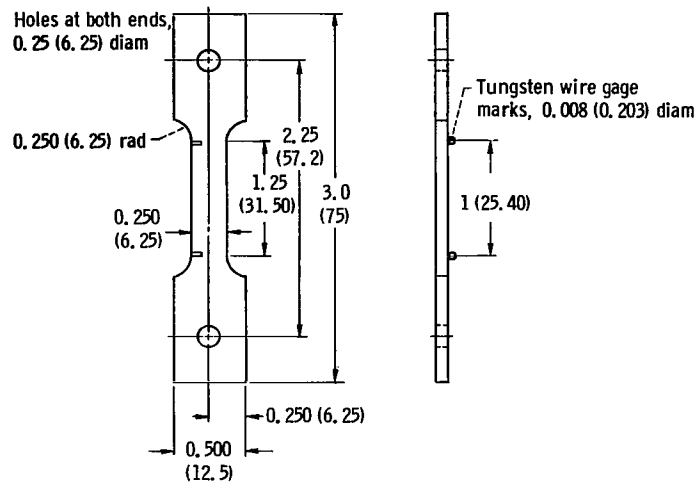


Figure 1. - High-temperature creep-rupture sheet specimen. Dimensions are in inches (mm).

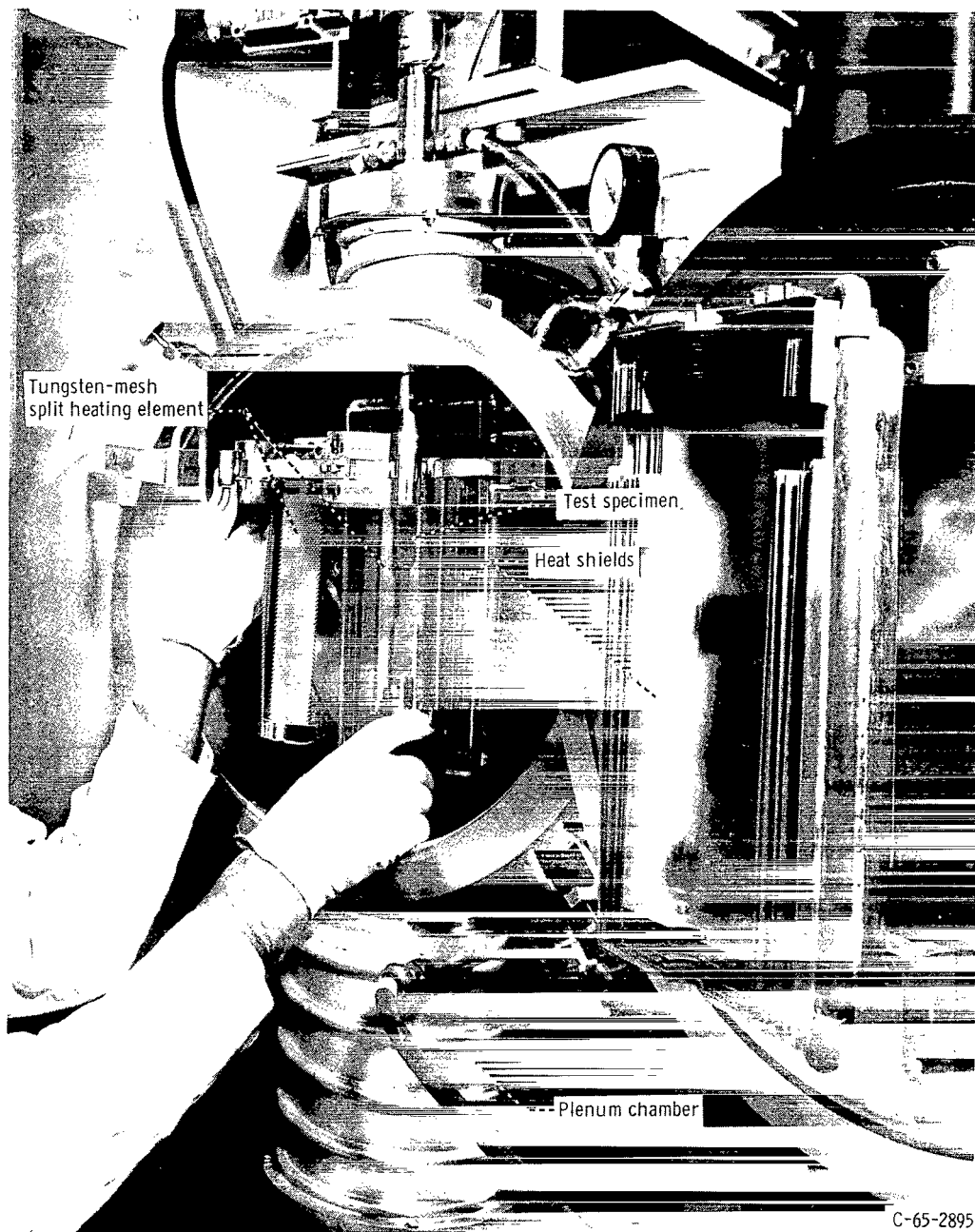


Figure 2. - High-temperature creep-rupture furnace.



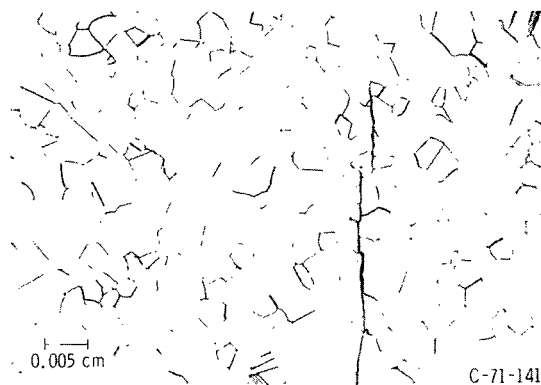
(a) As-received specimen (~2400° F (1316° C)) anneal during fabrication.



(b) Annealed at 2900° F (1593° C).

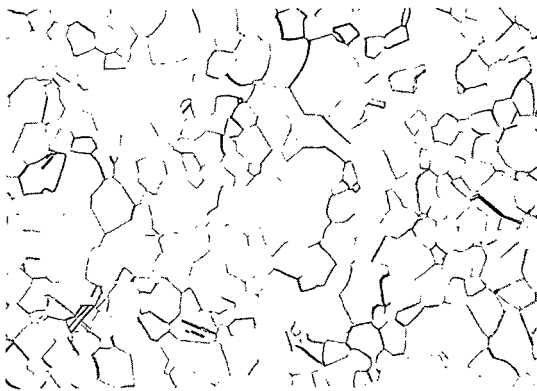


(c) Annealed at 3200° F (1760° C).

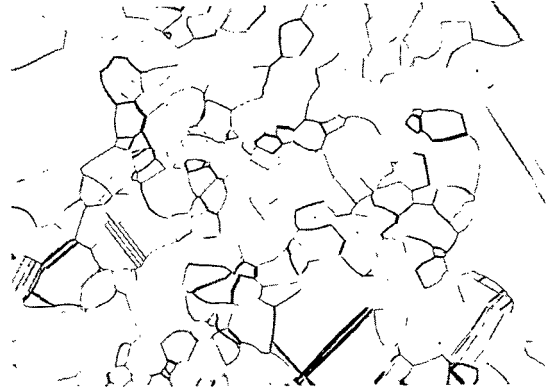


(d) Annealed at 3400° F (1871° C).

Figure 3. - Microstructure of powder metallurgy tungsten - 25-atomic-percent-rhenium - 30-atomic-percent-molybdenum sheet after various 2-hour anneals. Sheet was bend tested at room temperature after annealing, resulting in twinning. X250. Etchant, Murikami's reagent.



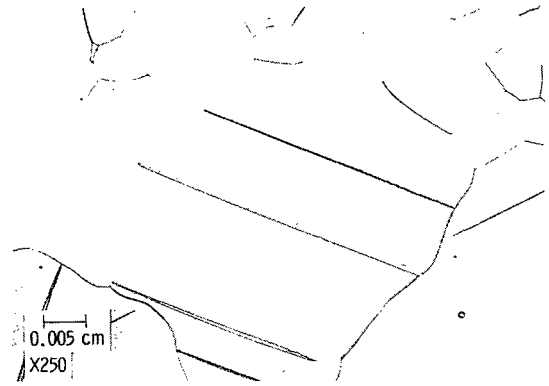
(e) Annealed at 3600° F (1982° C).



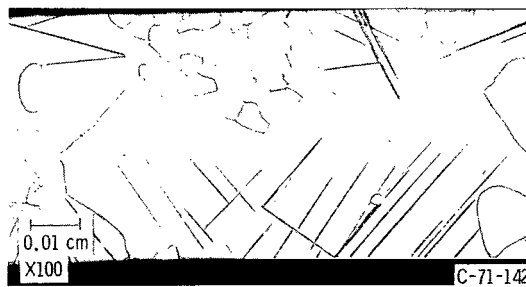
(f) Annealed at 3700° F (2038° C).



(g) Annealed at 3800° F (2093° C).

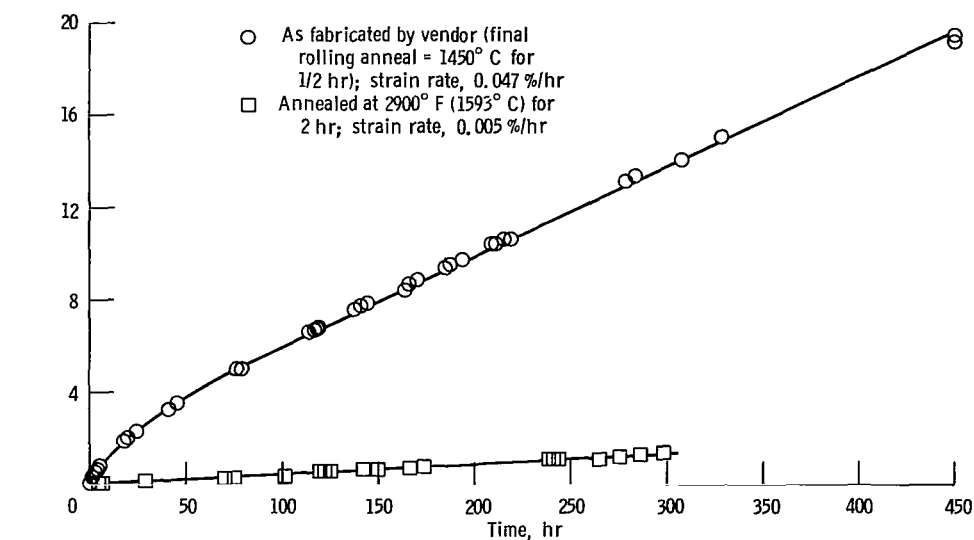


(h) Annealed at 4000° F (2204° C).

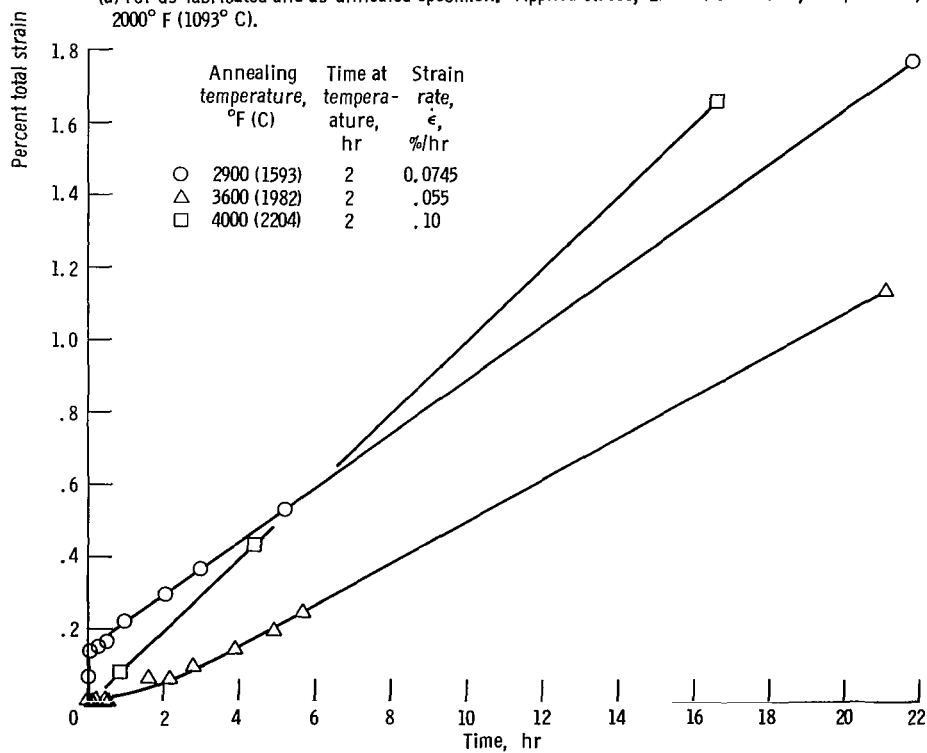


(i) Annealed at 4000° F (2204° C).

Figure 3. - Concluded.

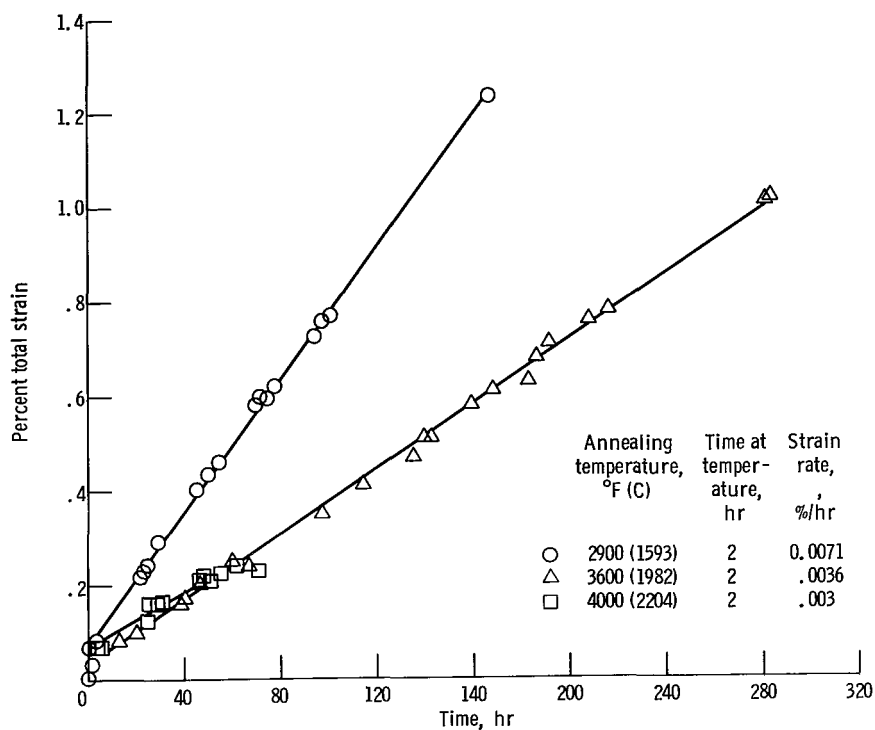


(a) For as-fabricated and as-annealed specimen. Applied stress, 20 ksi (138 MN/m²); temperature, 2000° F (1093° C).



(b) For three annealing treatments. Applied stress, 12 ksi (82.7 MN/m²); temperature, 2400° F (1316° C).

Figure 4. - Creep curves for tungsten - 25-atomic-percent- rhenium - 30 atomic-percent- molybdenum.



(c) For three annealing treatments. Applied stress, 4 ksi (27.6 MN/m²); temperature, 2400° F (1316° C).

Figure 4. - Concluded.

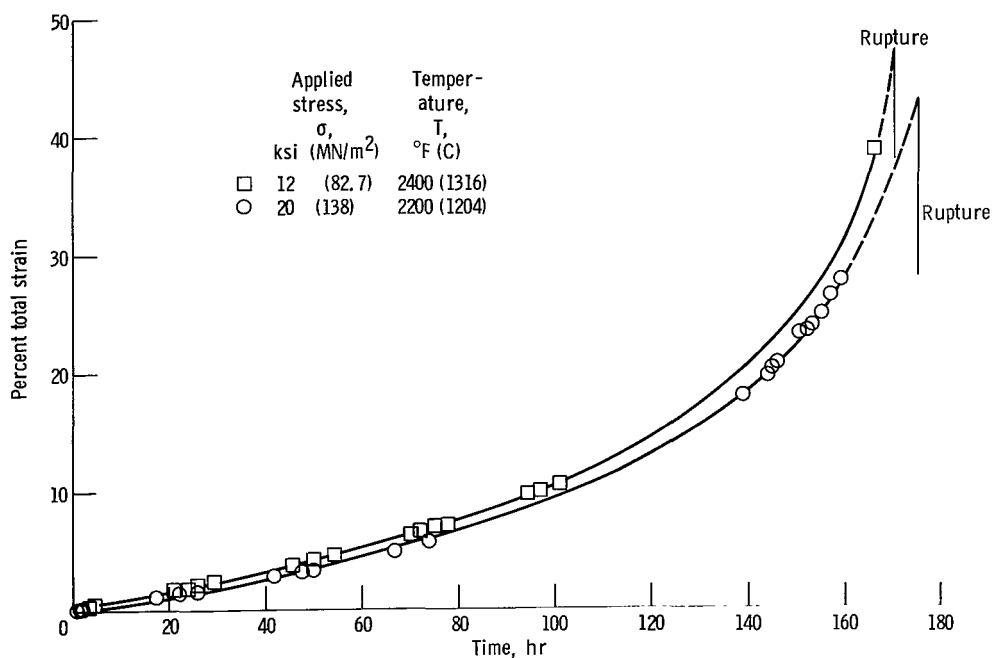


Figure 5. - Creep curves for tungsten - 25-atomic-percent- rhenium - 30-atomic-percent molybdenum annealed at 2900° F for 2 hours.

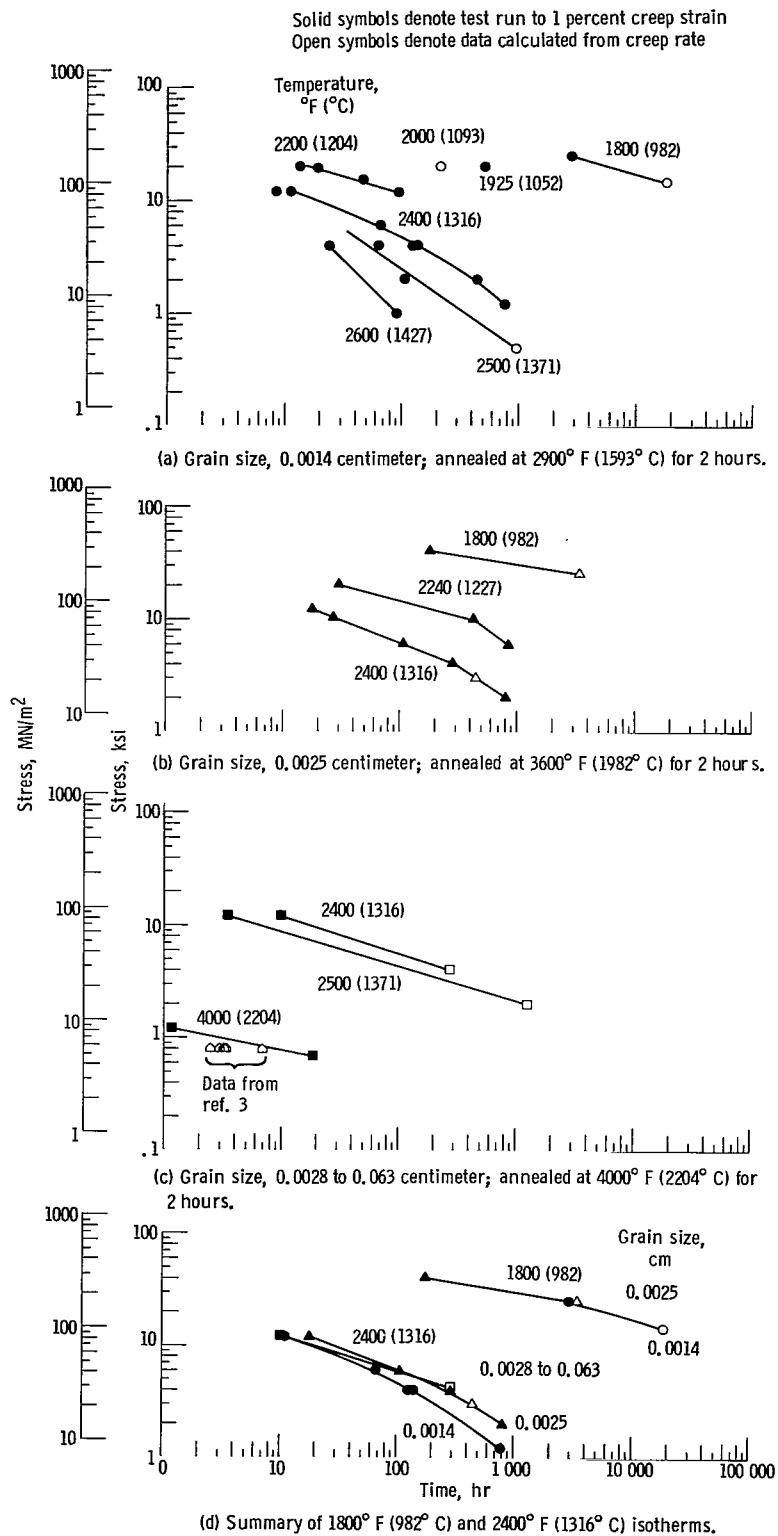
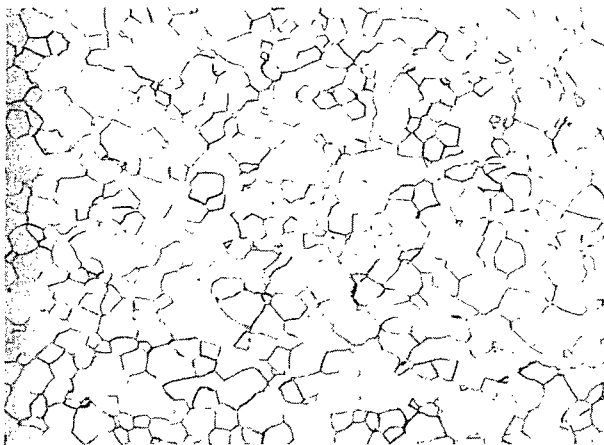
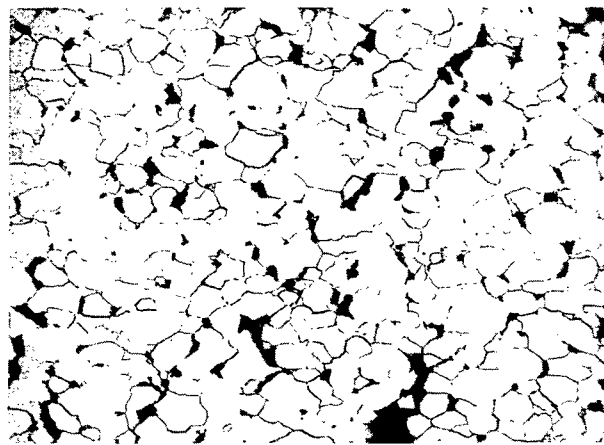


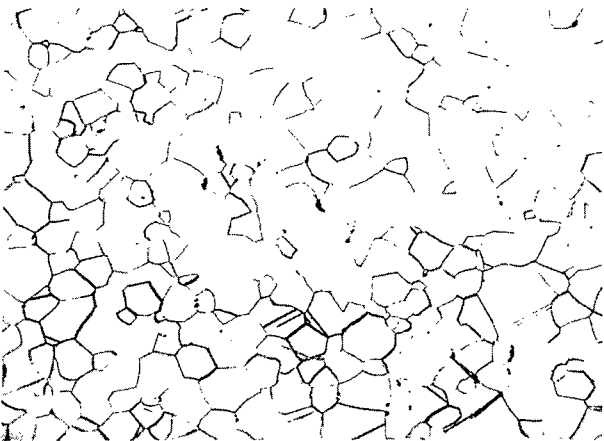
Figure 6. - Time to reach 1 percent total strain in powder metallurgy tungsten - 25-at.-%-rhenium - 30-at.-%-molybdenum sheet.



(a) Annealed at 2900° F (1593° C) for 2 hours. Creep test conditions: applied strain, 20 ksi (138 MN/m²); temperature, 2200° F (1204° C); percent total strain, 2.3.



(b) Annealed at 2900° F (1593° C) for 2 hours. Creep test conditions: applied strain, 12 ksi (82.7 MN/m²); temperature, 2400° F (1316° C); percent total strain, 42.



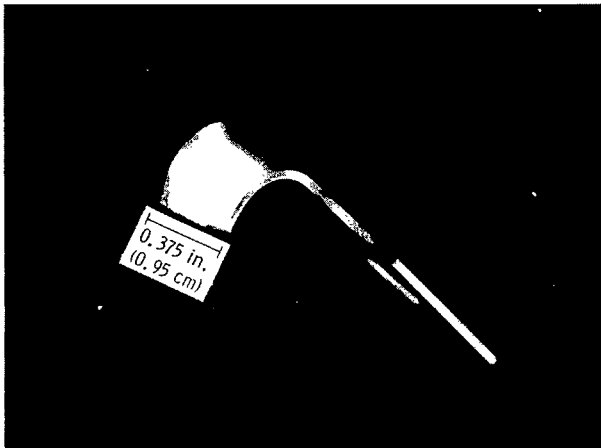
(c) Annealed at 3600° F (1982° C) for 2 hours. Creep test conditions: applied step-load stresses, 12, 10, 4, 2, and 3 ksi (82.7, 69, 27.6, 13.8, and 20.7 MN/m²); temperature, 2400° F (1316° C); percent total strain, 5.9.



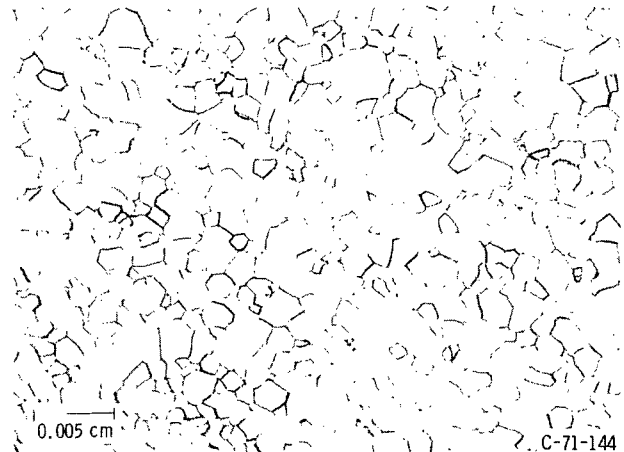
(d) Annealed at 4000° F (2204° C) for 2 hours. Creep test conditions: applied stresses, 0.69 and 1.2 ksi (4.8 and 8.3 MN/m²); temperature, 4000°, 3005°, and 2600° F (2204°, 1651°, and 1427° C); percent total strain, ~7.

C-71-143

Figure 7. - Test sections of creep test specimens showing grain boundary separation. Creep test loading axis was horizontal in the photomicrographs. Etchant, Murakami's reagent.



(a) Portion of creep test specimen used for room-temperature bend test.



(b) Microstructure of creep test section. Etchant, Murakami's reagent.

Figure 8. - Tungsten - 25-atomic-percent-rhenium - 30-atomic-percent molybdenum creep test specimen, annealed for 533 hours and creep tested at 1800° F (982° C) for 4792 hours, and bent 125° at room temperature over a 4T radius.

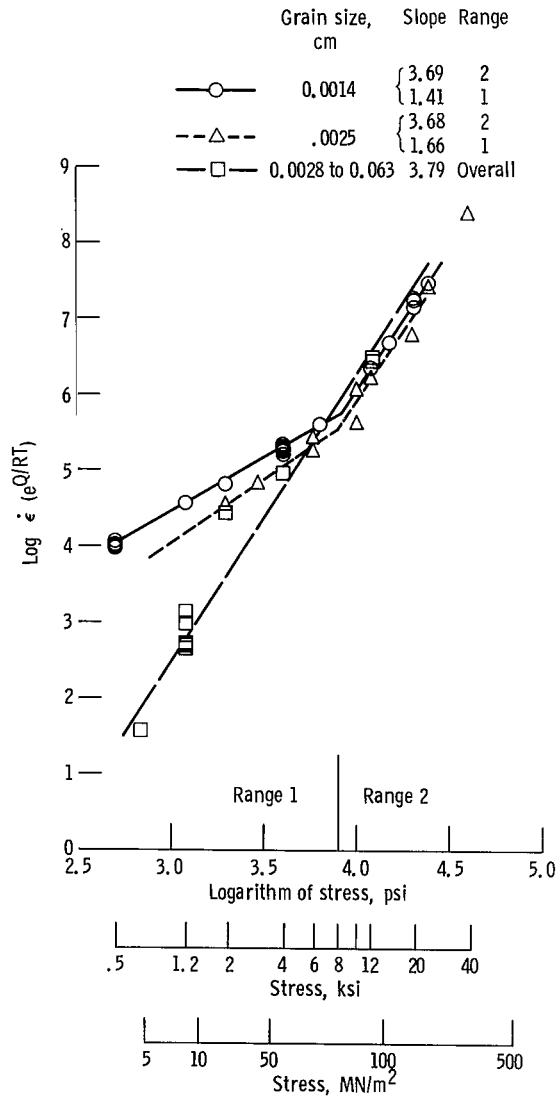


Figure 9. - Stress dependence of temperature compensated minimum creep rate for powder metallurgy tungsten - 25-atomic-percent-rhenium - 30-atomic-percent-molybdenum sheet.

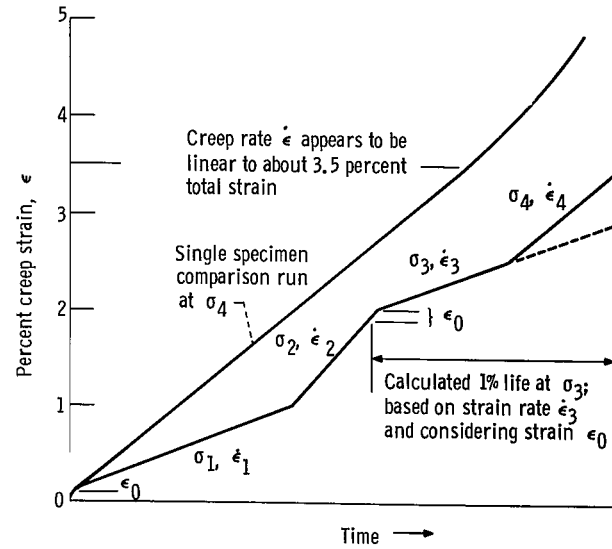


Figure 10. - Step loaded creep curve compared with single specimen creep curve.